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<p>(54) Title: TABLETOP SWEETENER COMPOSITIONS COMPRISING SWEETENER WITH EXTREMELY HIGH POTENCY (57) Abstract A novel tabletop sweetener product is disclosed. The tabletop sweetener contains N-[N-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine 1-methyl ester to provide some or all of the sweetness of the product. The sweetener may also contain other sweeteners and agents to provide bulking.</p>		

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TITLE

TABLETOP SWEETENER COMPOSITIONS COMPRISING SWEETENER
WITH EXTREMELY HIGH POTENCY

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BACKGROUND OF THE INVENTION

Field of the Invention

- 10 The present invention relates to novel tabletop sweetener compositions,
particularly tabletop sweeteners having reduced calorie contribution from sugars.

Description of the Prior Art

- 15 The market for sweetener products containing high intensity sweeteners is very
significant. Products such as Equal® sweetener have high levels of sales at
retail and at food service. These products are commonly referred to as tabletop
sweeteners. These types of products are found in packet form, in tablet form, in
spoon-for-spoon equivalents to sugar, and in liquid form. In each of these
20 product forms, the high intensity of the sweetener presents challenges to coming
up with a product in which the sweetness from the sweetener is uniform
throughout the product. For example, L-aspartyl-L-phenylalanine 1-methyl
ester, the sweetening ingredient in Equal® sweetener, has a potency upward of

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200 times the potency of sugar or high fructose corn syrup. Thus, tabletop sweeteners conventionally include one or more agents which add to the bulk of the tabletop sweetener product. Common agents include dextrose, maltodextrin, lactose, and combinations thereof. Such agents dissolve quickly and have tastes
5 which improve or do not interfere with the taste of the high intensity sweetener.

U.S. Patent No. 5,480,668 discloses a series of novel sweeteners having sweetness potency of up to 8,000 time the sweetening potency of sucrose on a weight basis; the most preferred sweetener covered therein shares structural
10 similarities with aspartame and therefore may become a revolutionary sweetener due to the step change in potency.

SUMMARY OF THE INVENTION

15 It has been discovered that tabletop sweetener products can be produced using the high intensity sweetener, N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester (neotame) to provide some or all of the sweetness desired in such tabletop sweetener. The tabletop sweeteners of this invention will generally contain N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine
20 1-methyl ester in an amount from about 0.005%(w/w) to about 1.2%(w/w) of the tabletop sweetener. Such tabletop sweeteners have been found to have clean, sweet taste profiles, with the usage level being extremely low.

DETAILED DESCRIPTION OF THE INVENTION

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The tabletop sweeteners of this invention are sweetened by the addition of the sweetener N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester. This sweetening ingredient has been found to have extremely high potency compared to sucrose while having a clean, sweet taste like that of
30 sucrose in tabletop sweeteners. This combination is crucial to consumer acceptance of tabletop sweeteners. However, the extremely high potency brings new challenges to the manufacture of tabletop sweeteners. Content uniformity

and uniform dissolution are even more crucial given the extremely small amount of the sweetener needed to provide the desired sweetness.

Thus for non-liquid tabletop sweeteners, i.e., solids, powders and granulated forms, the N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester sweetening ingredient should be used with an agent selected to provide bulk while not detracting from the clean sweet taste of the sweetening ingredient. Suitable bulking agents include, but are not limited to, Unidex brand mixture of 97% dextrose and 3% maltodextrin available from CPC International, dextrose, maltodextrin 100 (10 DE), maltodextrin 180 (18 DE), maltodextrin 40 (5 DE), corn syrup solids (20 DE), corn syrup solids (36 DE), sorbitol, erythritol, maltitol, lactitol, isomalt, maltose, tagatose, lactose, inulin, polyols, polydextrose, cellulose and cellulose derivatives, such as microcrystalline cellulose and carboxymethyl cellulose, and mixtures thereof. Additionally, table sugar (sucrose) or other caloric sweeteners such as crystalline fructose can be used as a bulking agent, as they provide good content uniformity while adding minimal calories, as the sucrose or other caloric sweetener is required only in the amounts necessary for bulk.

In addition, the sweetening ingredient and bulking agent may be further combined with a flow agent to assist in content uniformity and uniform dissolution. An exemplary flow agent is cream of tartar. The tabletop sweeteners of this invention may also include an anti-caking agent such as calcium silicate.

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Conventional caloric sweeteners or other high intensity sweeteners also may be used in a suitable tabletop sweetener product in combination with N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester. Such high intensity sweeteners include, but are not limited to, aspartame, acesulfame salts (e.g., acesulfame-K), sucralose, saccharin, alitame, cyclamates, stevia derivatives, thaumatin, glycyrrhizins (e.g., mono-, di- and tri-ammoniated forms) and neohesperidin dihydrochalcone (NHDC). Such conventional caloric

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sweeteners include, but are not limited to sucrose (in liquid or granular form) high fructose corn syrup, invert sugar, dextrose, glucose, crystalline fructose, high conversion corn syrup and polyol sugar alcohols.

- 5 To the extent other safe and suitable high intensity sweeteners like aspartame, acesulfame-K, sucralose, saccharin, alitame, cyclamates, stevia derivatives, thaumatin, glycyrrhizins, NHDC and/or conventional caloric sweeteners are used with the sweetening ingredient, the amount of the N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester can be reduced based on the amount of
- 10 such sweetener used and the potency of such sweeteners at the desired level of use. Similarly, combinations of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and table sugar (sucrose) can be used in a wide range of ratios. For example, 0.7 mg of N-[N-(3,3-dimethylbutyl)-L-aspartyl]-L-phenylalanine 1-methyl ester could be used in a packet in combination with 4
- 15 g of granulated sucrose to produce a tabletop sweetener equivalent in sweetness to 3 teaspoons of sugar, while having 16 calories as opposed to 48 for three teaspoons of table sugar. Such a product will have a taste like the taste of pure table sugar. Amounts even less than 0.3 mg may be used if it is desired to use larger amounts of any of the high intensity or other sweeteners described above.
- 20 It may be possible in blends to use amounts of N-[N-(3,3-dimethylbutyl)-L-aspartyl]-L-phenylalanine 1-methyl ester as low as 0.005% (w/w) in the product.

- Similarly, the flexibility of sweetener usage levels of combinations with other high intensity sweeteners is increased due to this compound's extremely high
- 25 potency. The use of the sweetener alone or in combination with other sweeteners or bulking agents can be adjusted in order to tailor the taste of the tabletop sweetener product to a specific end use. The desired product may vary if a specific food application is being targeted. For example, coffee, tea, cereal and fruit each have different taste characteristics which may affect the desired
- 30 level of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester when added via a tabletop sweetener formulation. The taste profile of the

sweetener can be modeled after the profile of the taste of table sugar in each of these individual products.

- Beverages, such as, for example, automatic drip coffee, instant coffee, iced tea
5 brewed from tea bags, and instant tea may be sweetened with the tabletop sweeteners of this invention.

- The forms of tabletop sweeteners which may successfully use N-[N-3,3-dimethylbutyl-L- α -aspartyl]-L-phenylalanine 1-methyl ester include sachets or
10 packets including the sweetener in powder or granular form, tablets, liquid sweeteners, and spoon for spoon. The sweetener may be measured into jars, pouches, pockets, bags, or the like. One skilled in the art will understand that the sweetening potency delivered by a tabletop sweetener can be varied depending on the end use and consumer preference. For example, the
15 formulations may be prepared so that one sweetener packet, one tablet, or a specified aliquot of the liquid sweetener in about 240 ml (one cup) of coffee or tea is approximately equivalent to the sweetness of one to three teaspoons of sucrose.

- 20 The sweetener also provides flexibility so that products may be formulated for other targeted uses, for example, a baking formulation having additional protecting agents such as encapsulants. Other forms will be readily apparent to those skilled in the tabletop sweetener art. The tabletop sweetener may be produced by combining the sweetening ingredient with the other product
25 ingredients via conventional methods.

- If the tabletop sweetener is a liquid sweetener then N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester is combined with a liquid carrier. The liquid carrier may be water, alcohol, polyol, glycerin base or citric acid base
30 dissolved in water. Preferably the carrier is a water/alcohol mixture, and most preferably a water/ethanol, water/sorbitol or water/ethanol/sorbitol mixture. The liquid sweetener may contain N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-

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phenylalanine 1-methyl ester in an amount from about 0.005% (w/w) to about 1.2 % (w/w) of the liquid sweetener. The general acceptable amount of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester contained in the liquid sweeteners is from about 0.025% (w/w) to about 0.500% (w/w). The preferred amount of the compound will be from about 0.046% (w/w) to about 0.140% (w/w). More preferably, the amount will be from about 0.081% (w/w) to about 0.105% (w/w).

In a powdered tabletop form, N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester is generally used with a bulking agent. Preferably the bulking agent is a mixture of dextrose and maltodextrin such as provided by the Unidex brand mixture of 97% dextrose and 3% maltodextrin. The powdered combination of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and a bulking agent such as Unidex may be packaged into packets. The general acceptable amount of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester contained in these packets is from about 0.025% (w/w) to about 0.500% (w/w). The preferred amount of the compound will be from about 0.046% (w/w) to about 0.140% (w/w). More preferably, the amount will be from about 0.081% (w/w) to about 0.105% (w/w). Again, lower amounts, (e.g. 0.005% w/w), of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester may be used when natural or other high intensity sweeteners are employed.

Any form of neotame may be used in the tabletop sweetener of this invention. For example, salts and metal complexes of neotame may be used, such as disclosed in U.S. Patent Application No. 09/146,963, U.S. Patent Application No. 09/146,964, U.S. Patent Application No. 09/148,134, U.S. Patent Application No. 09/146,965, all filed September 4, 1998, and all of which are incorporated by reference herein. Other exemplary forms of neotame that may be useful in this invention include cyclodextrin/neotame complexes such as disclosed in U.S. Provisional Patent Application No. 60/100,867 and cocrystallized neotame disclosed in U.S. Patent Application No. 09/154,568,

both filed September 17, 1998, and the disclosure of both of which are incorporated by reference herein.

The invention can be more readily understood by referring to the examples set forth below. The Examples which follow are intended as an illustration of certain preferred embodiments of the invention and no limitation of the invention is implied.

EXAMPLES

10

Example 1: Tabletop packets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and the bulking agent Unidex (97% dextrose and 3% maltodextrin; available from CPC International).

15 Approximately 1 g sweetener packets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and the bulking agent, Unidex, were prepared. N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and Unidex were dry blended and the resulting dry blend was packaged into 1 gram packets. Each packet contained approximately 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and
20 (3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and approximately 99.907% (w/w) Unidex (97% dextrose with 3% maltodextrin).

The packets were used to sweeten Folgers brand Aroma Roasted coffee, black and whitened with 2% milk. When mixed into approximately 240 ml (one cup)
25 of the coffee, the packets yielded an acceptably sweet product. One packet is approximately equivalent to the sweetness of two teaspoons of sucrose.

Various N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended with Unidex compositions were prepared and evaluated using coffee.
30 The general acceptable amount of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester in the packet composition is from about 0.025% (w/w) to about 0.500% (w/w). The preferred amount of N-[N-(3,3-

dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester is from about 0.046% (w/w) to about 0.140% (w/w). More preferably, the amount will be from about 0.081% (w/w) to about 0.105% (w/w).

- 5 Examples 2-6: Tabletop packets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended with other sweeteners and the bulking agent Unidex.

Tabletop packets were prepared in a manner substantially similar to Example 1,
10 with the exception that the tabletop packets of Examples 2-6 contained Unidex and N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended with another sweetener, i.e., either aspartame (APM), acesulfame-K (Ace-K), saccharin, sucralose, or sucrose. The various compositions were evaluated by their use in coffee, black and whitened with 2% milk.

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Example 2: N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended APM.

Various tabletop packets containing Unidex as the bulking agent and differing
20 concentrations of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and APM were prepared. A low level of one sweetener was combined with a high level of the other sweetener. The general range for adding N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended with APM is from about 0.005% (w/w) to about 0.500% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about
25 0.01% (w/w) to about 20% (w/w) APM. The preferred range is from about 0.0115% (w/w) to about 0.105% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about 0.426% (w/w) to about 4.16% (w/w) APM. The most preferred range is from about 0.032% (w/w) to about
30 0.063% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about 1.30% (w/w) to about 2.50% (w/w) APM.

Example 3: N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended Ace-K.

Various tabletop packets containing Unidex as the bulking agent and differing
5 concentrations of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine
1-methyl ester and Ace-K were prepared. A low level of one sweetener was
combined with a high level of the other sweetener. The general range for
adding N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester
blended with Ace-K is from about 0.005% (w/w) to about 0.500% (w/w) N-[N-
10 (3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about
0.01% (w/w) to about 28.4% (w/w) Ace-K. The preferred range is from about
0.0184% (w/w) to about 0.112% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-
L-phenylalanine 1-methyl ester and from about 0.530% (w/w) to about 4.77%
(w/w) Ace-K. The most preferred range is from about 0.041% (w/w) to about
15 0.0735% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine
1-methyl ester and from about 1.39% (w/w) to about 2.98% (w/w) Ace-K.

Example 4: N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl
ester blended Saccharin.

20

Various tabletop packets containing Unidex as the bulking agent and differing
concentrations of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine
1-methyl ester and saccharin were prepared. A low level of one sweetener was
combined with a high level of the other sweetener. The general range for
25 adding N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester
blended with saccharin is from about 0.005% (w/w) to about 0.500% (w/w) N-
[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from
about 0.01% (w/w) to about 16.1% (w/w) saccharin. The preferred range is
from about 0.0115% (w/w) to about 0.105% (w/w) N-[N-(3,3-dimethylbutyl)-L-
30 α -aspartyl]-L-phenylalanine 1-methyl ester and from about 0.375% (w/w) to
about 3.375% (w/w) saccharin. The most preferred range is from about
0.0324% (w/w) to about 0.063% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-

L-phenylalanine 1-methyl ester and from about 1.05% (w/w) to about 2.03% (w/w) saccharin.

Example 5: N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended sucralose.

Various tabletop packets containing Unidex as the bulking agent and differing concentrations of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and sucralose were prepared. A low level of one sweetener was combined with a high level of the other sweetener. The general range for adding N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended with sucralose is from about 0.005% (w/w) to about 99.9% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about 0.01% (w/w) to about 99.995% (w/w) sucralose. The preferred range is from about 0.0184% (w/w) to about 0.0840% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about 1.0% (w/w) to about 4.50% (w/w) sucralose. The most preferred range is from about 0.0162% (w/w) to about 0.0735% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about 1.314% (w/w) to about 4.5% (w/w) sucralose.

Example 6: N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended sucrose.

Various tabletop packets containing Unidex as the bulking agent and differing concentrations of N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and sucrose were prepared. A low level of one sweetener was combined with a high level of the other sweetener. The general range for adding N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended with sucrose is from about 0.005% (w/w) to about 0.5% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about 5% to about 99.995% sucrose. If sucrose is used as a bulking agent, the

- preferred range for adding N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester blended with sucrose is from about 0.035% (w/w) to about 0.127% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about 99.873% (w/w) to about 99.965% (w/w) sucrose.
- 5 The most preferred range is from about 0.069% (w/w) to about 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and from about 99.895% (w/w) to about 99.919% (w/w) sucrose.

- Example 7: Tabletop packets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and the bulking agent Erythritol.
- 10

- Approximately 1 g sweetener packets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and Erythritol (a bulking agent) were prepared. N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester, Erythritol, and the flow agent were dry blended, and the resulting dry blend was packaged into 1 gram packets. Each packet contained approximately 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and 99.907% (w/w) Erythritol.
- 15

- 20 The packets were used to sweeten Folgers brand Aroma Roasted coffee, black and whitened with 2% milk, and iced tea, made from Lipton Brisk Tea Bags. When mixed into approximately 240 ml (one cup) of the coffee and tea, the packets yielded an acceptably sweet product.

- 25 Example 8: Tabletop packets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester, the bulking agent Lactose and the flow agent silicon dioxide.

- Approximately 1 g sweetener packets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester, Lactose (a bulking agent), and silicon dioxide (a flow agent) were prepared. N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester, Lactose, and the flow agent were dry blended,
- 30

and the resulting dry blend was packaged into 1 gram packets. Each packet contained approximately 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester, 99.507% (w/w) Lactose, and 0.4% (w/w) flow agent. The packets were used to sweeten Folgers brand Aroma Roasted coffee, black and whitened with 2% milk, and iced tea, made from Lipton Brisk Tea Bags. When mixed into approximately 240 ml (one cup) of the coffee and tea, the packets yielded an acceptably sweet product.

Example 9: Tablets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester.

The constituents used to formulate the Tablets of Example 9 are shown in Table 1.

Table 1
Example 9 formulation
Tablets containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester

	Ingredient	Formula (%w/w)	Batch Weight (g)
20	Glycine (Chattem Chemical, Chattanooga, TN)	65.50	327.5
	Maltrin M-100 (Coin Products, Summit/Argo, IL)	3.0	15.0
	L-Leucine (Ajinomoto, Tokyo, Japan)	1.750	8.750
25	Avicel PH 102 (FMC, Philadelphia, PA)	2.750	13.750
	N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester	0.675	3.375
	Unidex (CPC Int'l., Summit/Argo, IL)	26.325	131.625
30	Total	100.0	500.0

The Tablets of Example 9 were made by putting all of the above ingredients into a Hobart mixer and mixing for approximately 15 minutes. Ten 0.85 gram portions of the above dry mix were weighed. One of the 0.85 gram portions

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was added to a Carver Laboratory Press, Hydraulic Unit Model 3912. A tablet was made by applying 600 psi of pressure. The resulting tablet was examined to ensure that no loose particles remained on the edge. The remaining tablets were made accordingly.

5

Whitened coffee was prepared by mixing 2,400 ml of Folgers Aroma Roasted coffee with 450 ml of 2% milk. One 0.85 gram tablet was added to approximately 240 ml of the whitened coffee and dissolved. The dissolved tablet yielded acceptably sweet coffee. One 0.85 g tablet is approximately

10 equivalent to the sweetness of two teaspoons of sucrose.

Example 10: Liquid Sweetener containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester.

15 The constituents used to formulate the Liquid Sweetener of Example 10 are shown in Table 2.

Table 2
Example 10 Formulation
Liquid Sweetener containing N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester

5	Ingredient	Formula (%w/w)	Batch Weight (g)
	N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester	0.200	0.60
	Sodium Benzoate	0.100	0.30
10	Potassium Sorbate	0.100	0.30
	10% Phosphoric Acid	1.050	3.150
	Sorbitol, 70% solution	15.0	45.0
	Ethanol	0.500	1.50
	Water	83.050	249.150
15	Total	100.0	300.0

The Liquid Sweetener of Example 10 was made by first weighing out the water. The N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester was premixed with ethanol. Then the N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester/ethanol solution, sodium benzoate, potassium sorbate were added to the water and mixed for approximately 5 minutes, until all dry ingredients were dissolved. The N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester also may be directly added to the water with the other dry ingredients, eliminating the need for ethanol in the formulation.

25 The 70% Sorbitol Solution was then added to the water solution. The pH of the resulting solution was adjusted to approximately 4.3 to 4.4 using the 10% phosphoric acid solution.

30 The resulting Liquid Sweetener was added to approximately 240 ml of water in a 0.6 gram aliquot. The water was found to be acceptably sweet. One 0.6 gram

aliquot of liquid sweetener is approximately equivalent to the sweetness of two teaspoons of sucrose.

Example 11: Spoon for Spoon Tabletop Sweetener containing N-[N-(3,3-
5 dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester.

A spoon for spoon tabletop sweetener containing 0.1% (w/w) N-[N-(3,3-
dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and 99.9%
maltodextrin was prepared. The sweetener was prepared by mixing 50 kg of a
10 maltodextrin/N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl
ester (99.9:0.1 wt. ratio) blend with 50 kg of water heated to 65°C (150°F).
The mixture was then spray dried after injection of CO₂ into the liquid stream to
provide a product having an effective bulk density of about 0.1 g/cm³. This
formulation provided a sweetness potency equal to about one teaspoon of
15 sucrose.

WHAT IS CLAIMED IS:

1. A tabletop sweetener comprising N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester in an amount of from about 0.005% (w/w) to about 1.2% (w/w) of said tabletop sweetener.
2. The tabletop sweetener of claim 1, wherein said sweetener is in a powder or granulated form and the N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester is present in an amount of from about 0.025% (w/w) to about 0.5% (w/w) of said tabletop sweetener.
3. The tabletop sweetener of claim 2, wherein said N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester is present in an amount of from about 0.046% (w/w) to about 0.14% (w/w) of said tabletop sweetener.
4. The tabletop sweetener of claim 3, wherein said N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester is present in an amount of from about 0.081% (w/w) to about 0.105% (w/w) of said tabletop sweetener.
5. The tabletop sweetener of claim 1, wherein said sweetener is selected from the forms consisting of powder form, pocket form, granular form, tablet form, and liquid form.
6. The tabletop sweetener of claim 5, wherein said sweetener in powder or granular form is packaged in packets containing the equivalent sweetness of from about one to about three teaspoons of sucrose.
7. The tabletop sweetener of claim 6, wherein said packet contains the equivalent sweetness of about two teaspoons of sucrose.

8. The tabletop sweetener of claim 5, wherein said sweetener in tablet form contains the equivalent sweetness of from about one to about three teaspoons of sucrose per tablet.

9. The tabletop sweetener of claim 8, wherein said tablet contains the equivalent sweetness of about two teaspoons of sucrose.

10. The tabletop sweetener of claim 5, wherein said sweetener in liquid form contains the equivalent sweetness of from about one to about three teaspoons of sucrose per approximately 0.6 gram aliquot of liquid.

11. The tabletop sweetener of claim 10, wherein said approximately 0.6 gram aliquot of liquid contains the equivalent sweetness of about two teaspoons of sucrose.

12. The tabletop sweetener of claim 1, wherein said tabletop sweetener comprises at least one sweetener selected from the group consisting of aspartame, acesulfame-K, sucralose, saccharin, alitame, cyclamates, stevia derivatives, thaumatin, glycyrrhizins, neohesperidin dihydrochalcone, polyol sugar alcohols, sucrose, high fructose corn syrup, invert sugar, dextrose, crystalline fructose, high conversion corn syrup and mixtures thereof.

13. The tabletop sweetener of claim 1, wherein said tabletop sweetener comprises sucrose.

14. The tabletop sweetener of claim 1, wherein said tabletop sweetener comprises at least one bulking agent selected from the group consisting of dextrose, maltodextrin 100 (10 DE), maltodextrin 180 (18 DE), maltodextrin 40 (5 DE), corn syrup solids (20 DE), corn syrup solids (36 DE), sorbitol, erythritol, maltitol, lactitol, isomalt, maltose, tagatose, lactose, fructose, inulin, polyols, polydextrose, and cellulose and cellulose derivatives, and mixtures thereof.

15. The tabletop sweetener of claim 2, further comprising a bulking agent comprising a mixture of dextrose and maltodextrin.
16. The tabletop sweetener of claim 15, wherein said bulking agent is about 97%(w/w) dextrose and about 3%(w/w) maltodextrin.
17. The tabletop sweetener of any one of claims 14-16, further comprising a flow agent.
18. The tabletop sweetener of claim 17, wherein said flow agent is selected from the group consisting of cream of tartar, calcium silicate, silicon dioxide, avicel and tricalcium phosphate.
19. The tabletop sweetener of any one of claims 14-16, further comprising an anti-caking agent.
20. The tabletop sweetener of claim 19, wherein said anti-caking agent is calcium silicate.
21. The tabletop sweetener of any one claims 14-16, further comprising at least one sweetener selected from the group consisting of aspartame, acesulfame-K, sucrose, sucralose, saccharin, alitame, cyclamates, stevia derivatives, thaumatin, glycyrrhizins, neohesperidin dihydrochalcone, polyol sugar alcohols, high fructose corn syrup, invert sugar, dextrose, glucose, crystalline fructose, high conversion corn syrup and mixtures thereof.
22. The tabletop sweetener of any one of claims 14-16, wherein said sweetener is in powder form and is packaged in packets.
23. The tabletop sweetener of claim 22, wherein said packet contains approximately 0.093% (w/w) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester and about 99.907% (w/w) bulking agent.

24. A substantially free-flowing solid tabletop sweetener comprising:

(a) N-[N-(3,3-dimethylbutyl)-L- α -aspartyl]-L-phenylalanine 1-methyl ester in an amount of from about 0.025%(w/w) to about 0.5%(w/w) of said tabletop sweetener:

(b) a bulking agent, selected from the group consisting of, dextrose, maltodextrin 100 (10 DE), maltodextrin 180 (18 DE), maltodextrin 40 (5 DE), corn syrup solids (20 DE), corn syrup solids (36 DE), sorbitol, erythritol, maltitol, lactitol, isomalt, maltose, tagatose, lactose, fructose, inulin, polyols, polydextrose, and cellulose and cellulose derivatives, and mixtures thereof;

(c) optionally, a sweetener, selected from the group consisting of aspartame, acesulfame-K, sucralose, saccharin, sucrose, alitame, cyclamates, stevia derivatives, thaumatin, neohesperidin dihydrochalcone, or polyol sugar alcohols;

(d) optionally, a flow agent; and

(e) optionally, an anti-caking agent.

25. The tabletop sweetener of claim 24, wherein said sweetener is in powder form and is packaged in packets.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US 98/26866

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: A23L 1/236

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5510508 A (NOFRE CLAUDE ET AL), 23 April 1996 (23.04.96)	1-25
	--	
X	US 5480668 A (CLAUDE NOFRE ET AL), 2 January 1996 (02.01.96)	1-25
	--	
P,X	WO 9839979 A1 (AJINOMOTO CO., INC.), 17 Sept 1998 (17.09.98)	1-25
	--	
A	WO 9215205 A1 (THE NUTRASWEET COMPANY), 17 Sept 1992 (17.09.92)	1-25
	--	

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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INTERNATIONAL SEARCH REPORT

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PCT/US 98/26866

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2292314 A (EAST WELLSUM INDUSTRIES (S) PTE. LTD), 21 February 1996 (21.02.96) --	1-25
A	EP 0681789 A1 (HOECHST AKTIENGESELLSCHAFT), 15 November 1995 (15.11.95) -- -----	1-25

SA 219082

INTERNATIONAL SEARCH REPORT

Information on patent family members

02/03/99

International application No.

PCT/US 98/26866

Patent document cited in search report			Publication date	Patent family member(s)	Publication date
US	55105+8	A	23/04/96	NONE	
US	5480668	A	02/01/96	AT 138935 T	15/06/96
				AU 664663 B	23/11/95
				AU 5468194 A	08/06/94
				BG 61609 B	30/01/98
				BG 99299 A	29/09/95
				CA 2139233 A	26/05/94
				CN 1038747 B	17/06/98
				CN 1090571 A	10/08/94
				CZ 9403319 A	18/10/95
				DE 69303032 D,T	02/10/96
				DK 669935 T	01/07/96
				EP 0669935 A,B	06/09/95
				ES 2091114 T	16/10/96
				FI 945451 A	22/12/94
				FR 2697844 A,B	13/05/94
				GR 3020164 T	30/09/96
				HU 72192 A	28/03/96
				HU 9403842 D	00/00/00
				IL 107551 A	16/08/98
				JP 2818032 B	30/10/98
				JP 8503206 T	09/04/96
				LT 1457 A	15/06/94
				LT 3142 B	31/01/95
				MD 960256 A	31/03/98
				NO 945090 A	30/12/94
				NZ 257870 A	26/03/96
				PL 306841 A	18/04/95
				SK 158694 A	10/05/95
				WO 9411391 A	26/05/94
				ZA 9308430 A	13/06/94
WO	9839979	A1	17/09/98	AU 4398097 A	29/09/98
				JP 10248520 A	22/09/98
				JP 10248521 A	22/09/98
WO	9215205	A1	17/09/92	AU 7497591 A	06/10/92
				CA 2082158 A,C	07/09/92
				EP 0533681 A,B	31/03/93
				GR 3020016 T	31/08/96
				US 5085876 A	04/02/92
				DK 533681 T	29/07/96
GB	2292314	A	21/02/96	CN 1110112 A	18/10/95
				GB 9416570 D	00/00/00
				WO 9527408 A	19/10/95
EP	0681789	A1	15/11/95	CZ 9501186 A	15/11/95
				DE 4416429 A	16/11/95
				FI 952202 A	11/11/95
				JP 8038099 A	13/02/96
				NO 951819 A	13/11/95

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